

**GUIDANCE DOCUMENT FOR ALTERNATIVE DIESEL  
FUELS PROPOSED AS “DROP-IN” FUELS TO  
DISPLACE DIESEL FUELS AS SPECIFIED BY ASTM  
SPECIFICATION D975**

**INTERIM REPORT  
TFLRF No. 451**

**by  
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Southwest Research Institute<sup>®</sup> (SwRI<sup>®</sup>)  
San Antonio, TX**

**for  
Patsy A. Muzzell  
U.S. Army TARDEC  
Force Projection Technologies  
Warren, Michigan**

**Contract No. W56HZV-09-C-0100 (WD21 Task 2.2)**

**UNCLASSIFIED: Distribution Statement A. Approved for public release**

**July 2014**

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**Approved by:**



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REPORT DOCUMENTATION PAGE				Form Approved OMB No. 0704-0188	
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<b>1. REPORT DATE (DD-MM-YYYY)</b> 07/21/2014		<b>2. REPORT TYPE</b> Interim Report		<b>3. DATES COVERED (From - To)</b> September 2012 – July 2014	
<b>4. TITLE AND SUBTITLE</b> Guidance Document for Alternative Diesel Fuels Proposed as “Drop-In” Fuels to Displace Diesel Fuels as Specified By ASTM Specification D975				<b>5a. CONTRACT NUMBER</b> W56HZV-09-C-0100	
				<b>5b. GRANT NUMBER</b>	
				<b>5c. PROGRAM ELEMENT NUMBER</b>	
<b>6. AUTHOR(S)</b> Westbrook, Steven R.				<b>5d. PROJECT NUMBER</b> SwRI 08.14734.21	
				<b>5e. TASK NUMBER</b> WD 21 Task 2.2	
				<b>5f. WORK UNIT NUMBER</b>	
<b>7. PERFORMING ORGANIZATION NAME(S) AND ADDRESS(ES)</b> U.S. Army TARDEC Fuels and Lubricants Research Facility (SwRI®) Southwest Research Institute® P.O. Drawer 28510 San Antonio, TX 78228-0510				<b>8. PERFORMING ORGANIZATION REPORT NUMBER</b> TFLRF No. 451	
<b>9. SPONSORING / MONITORING AGENCY NAME(S) AND ADDRESS(ES)</b> U.S. Army RDECOM U.S. Army TARDEC Force Projection Technologies Warren, MI 48397-5000				<b>10. SPONSOR/MONITOR'S ACRONYM(S)</b>	
				<b>11. SPONSOR/MONITOR'S REPORT NUMBER(S)</b>	
<b>12. DISTRIBUTION / AVAILABILITY STATEMENT</b> UNCLASSIFIED: Dist A Approved for public release; distribution unlimited					
<b>13. SUPPLEMENTARY NOTES</b>					
<b>14. ABSTRACT</b> This document provides guidance for consideration of requests to designate a new, renewable/alternative diesel fuel as a drop-in replacement. A suggested protocol is included along with information regarding each element of the protocol.					
<b>15. SUBJECT TERMS</b> Diesel fuel, renewable diesel fuel, alternative diesel fuel					
<b>16. SECURITY CLASSIFICATION OF:</b>			<b>17. LIMITATION OF ABSTRACT</b>	<b>18. NUMBER OF PAGES</b>	<b>19a. NAME OF RESPONSIBLE PERSON</b>
<b>a. REPORT</b> Unclassified	<b>b. ABSTRACT</b> Unclassified	<b>c. THIS PAGE</b> Unclassified	Unclassified	28	<b>19b. TELEPHONE NUMBER (include area code)</b>

Standard Form 298 (Rev. 8-98)  
Prescribed by ANSI Std. Z39.18

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## EXECUTIVE SUMMARY

Diesel engines and diesel fuels have developed in parallel over the past 100 years. Until recently, developments in both diesel engines and diesel fuels evolved slowly, in response to customer needs and commercial realities. Historically, diesel fuels have been produced from crude oils by refinery processes. A wide range of crude oils (from light, sweet crudes to very heavy, sour crudes and bitumens) have been used in a wide variety of refinery processes. The current specification for diesel fuel, ASTM D975 [1], is the outcome of this evolutionary process. With the concurrent development of diesel engines, fuels from conventional sources, produced by conventional refinery processes, are considered to be ‘fit-for-purpose’ in compression ignition, diesel engines if they comply with D975. Note that in recent years, even D975 for ‘conventional diesel fuels’ has had to be modified (with the addition of lubricity and conductivity requirements) to keep the fuels ‘fit-for-purpose’ as the chemistry of diesel fuels has changed to comply with new regulatory requirements.

However, as the industry has discovered recently, some fuels that may comply with the detailed requirements of D975 (principally Table 1 requirements) may not be fit-for-purpose in the full range of diesel engines in current service (generally engines produced over the past 25 years) due to significant differences in the chemistry of fuels produced from new sources, by new processes, containing new trace components, or contaminants that can lead to operational problems.

This document provides guidance relative to these alternative diesel fuels. Specifically, it contains information about the testing that could be required for new fuels, from new sources and produced by new processes, intended for use in compression ignition, diesel engines. The testing is meant to give confidence that a given alternative fuel will be ‘fit-for-purpose’. New fuels can be considered both as blending components to be used (up to a defined limit) with conventional diesel fuel, or as neat fuels to be used at 100%

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## **FOREWORD/ACKNOWLEDGMENTS**

The U.S. Army TARDEC Fuel and Lubricants Research Facility (TFLRF) located at Southwest Research Institute (SwRI), San Antonio, Texas, performed this work during the period September 2012 through June 2014 under Contract No. W56HZV-09-C-0100. The U.S. Army Tank Automotive RD&E Center, Force Projection Technologies, Warren, Michigan administered the project. Mr. Eric Sattler (RDTA-SIE-ES-FPT) served as the TARDEC contracting officer's technical representative. Patsy Muzzell of TARDEC served as project technical monitor.

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## ACRONYMS AND ABBREVIATIONS

ASTM – ASTM International

BXXX – Shorthand indication of percentage of biodiesel in a biodiesel blend

CFPP – ASTM D6371 Cold Filter Plugging Point

DOE – United States Department of Energy

EPA – United States Environmental Protection Agency

IRS – United States Internal Revenue Service

LTFT – ASTM D4539 Low Temperature Flow Test

SDS – Safety Data Sheet (also known as MSDS - Material Safety Data Sheet)

## 1.0 INTRODUCTION AND BACKGROUND

Diesel engines and diesel fuels have developed in parallel over the past 100 years. Until recently, developments in both diesel engines and diesel fuels evolved slowly, in response to customer needs and commercial realities. Historically, diesel fuels have been produced from crude oils by refinery processes. A wide range of crude oils (from light, sweet crudes to very heavy, sour crudes and bitumens) have been used in a wide variety of refinery processes. The current specification for diesel fuel, ASTM D975 [2], is the outcome of this evolutionary process. With the concurrent development of diesel engines, fuels from conventional sources, produced by conventional refinery processes, are considered to be ‘fit-for-purpose’ in compression ignition, diesel engines if they comply with D975. Note that in recent years, even D975 for ‘conventional diesel fuels’ has had to be modified (with the addition of lubricity and conductivity requirements) to keep the fuels ‘fit-for-purpose’ as the chemistry of diesel fuels has changed to comply with new regulatory requirements.

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This document provides guidance relative to these alternative diesel fuels. Specifically, it contains information about the testing that could be required for new fuels, from new sources and produced by new processes, intended for use in compression ignition, diesel engines. The testing is meant to give confidence that a given alternative fuel will be ‘fit-for-purpose’. New fuels can be considered both as blending components to be used (up to a defined limit) with conventional diesel fuel, or as neat fuels to be used at 100%

ASTM D975, as a specification for diesel fuel, was written to describe a particular cut from a barrel of petroleum oil. That was the assumption from the beginning of the specification and that

is the reason for most of the properties currently listed in D975, Table 1. There have been relatively few changes to the Table 1 requirements of the specification since it was first approved in 1949. However, as we have learned with biodiesel, the properties in D975 are not always sufficient to describe a fuel (or fuel component) that will be trouble free in normal use. For that reason, it is necessary to start from the assumption that any fuel for a diesel engine must not only burn in the engine but also not cause problems for the engine and the user. In other words, the fuel must be evaluated from the point of view of the intended use rather than the fuel itself.

## **2.0 DEFINITIONS OF TERMS USED IN THIS GUIDE**

Each of the terms discussed below is defined for the purposes of use in this guide. The definitions are as technically correct as can be given. If other definitions are found in common use, they are discussed and identified as such.

### **2.1 DIESEL FUEL (OR COMPRESSION IGNITION FUEL)**

Over the years, the term diesel fuel has grown to be a generic term for the liquid fuel used in a diesel engine. However, some slightly different definitions can be found. These differences tend to be the result of specific uses of the term. At the time of this writing, ASTM lists three separate definitions for diesel fuel as follows:

- *diesel fuel, n*—middle petroleum distillate fuel.
- *diesel fuel, n*—petroleum-based middle distillate fuel.
- *diesel fuel oil, n*—any petroleum liquid suitable for the generation of power by combustion in compression ignition (diesel) engines. [DISCUSSION—Different grades are characterized primarily by viscosity ranges and by minimum cetane numbers.]

While the differences in the above definitions are certainly minor, they do tend to cause some level of confusion in the marketplace. This is especially true when there is an attempt to introduce a “new diesel fuel” into the market.

In 2013, the ASTM subcommittee (D2.E0) with jurisdiction over the diesel fuel specification began work on definitions for the terms diesel fuel and alternative diesel fuel. For the purpose of this guide, the latest definition to be balloted by D2.E0 is the definition of diesel fuel that will be used. That definition is:

- *diesel fuel, n*-material used for the generation of power by combustion in compression ignition engines.

It is important to note that the definition used here contains no reference to the source or raw material of the fuel. This is intentional.

## 2.2 RENEWABLE DIESEL FUEL

DOE, EPA, and IRS have definitions, components of these definitions include:

- derived from biomass,
- Registration requirements for fuels and chemicals established by the Environmental Protection Agency under Section 211 of the Clean Air Act (42 U.S.C. 7545)
- Requirements of the ASTM International Standard Specifications D975 or D396
- Non-ester; not biodiesel

There are various processes for manufacture of these fuels:

- Hydrothermal Processing
- Hydroprocessing
- Indirect Liquefaction
- Biomass to liquid (via gasification/Fischer-Tropsch)
- Pyrolysis-Rapid Thermal Processing

The definition used for this guidance document is:

- *renewable diesel, n* – a class of liquid fuels, derived from biomass through any of a number of processes which convert the biomass to non-ester, liquid hydrocarbons, meeting the requirements of the appropriate ASTM fuel specification (i.e., D396, D975, etc.). [Discussion

- Renewable diesel must also meet United States Environmental Protection Agency registration requirements.]

### **2.3 DROP-IN FUEL**

A drop-in fuel is: fully compatible with current vehicles, fully compatible with the current fueling infrastructure, has approximately the same energy content as traditional, petroleum-derived fuels, and is composed of the same molecules as traditional fuels.

### **2.4 HYDROCARBONS**

Hydrocarbons are compounds that contain only carbon and hydrogen.

### **2.5 TRACE COMPONENTS**

Trace components are compounds found in a fuel at trace (parts per million) concentrations. They are in the fuel because they were in the raw material from which the fuel was made. Sulfur compounds in petroleum-derived diesel fuel are an example. Performance-enhancing additives and contaminants from the fuel delivery and storage system are not trace components.

## **3.0 FUEL PROPERTIES AND THEIR SIGNIFICANCE**

### **3.1 FUEL PROPERTIES REQUIRED BY ASTM D975**

ASTM specification D975 covers seven grades of diesel fuel oils suitable for various types of diesel engines. These grades are:

- Grade No. 1-D S15
- Grade No. 1-D S500
- Grade No. 1-D S5000
- Grade No. 2-D S15
- Grade No. 2-D S500
- Grade No. 2-D S5000
- Grade No. 4-D

The No. 1 grades are light middle distillate fuels for applications that require higher volatility than No. 2 grades. The No. 2 grades are middle distillate fuel for diesel engines, especially under conditions of varying speed and load. The SXXX designation denotes the maximum allowable sulfur content (in XXX ppm) for that grade. Grade No. 4-D is a heavy distillate fuel or a blend of distillate and residual oil. It is primarily used in low- and medium-speed diesel engines, predominantly under conditions of constant speed and load. The guidance in this document is related to the No.1 and No. 2 grades of diesel fuel.

The properties listed in “Table 1 Detailed Requirements for Diesel Fuel Oils” are flash point, water and sediment, distillation, viscosity, ash, sulfur, copper strip corrosion, cetane number, cetane index or aromaticity, cloud point (or LTFT/CFPP), carbon residue, lubricity, and conductivity. Some additional details/requirements are provided in the footnotes to the table. The reader is directed to the latest version of the specification to view these footnotes. At this writing, D975 also contains an allowance for up to 5% (volume) of biodiesel (meeting D6751). There is also a requirement that diesel fuels covered under D975 “shall be hydrocarbon oils,” with the exception of allowance for biodiesel and additives to enhance performance of the fuel. The resulting fuel must meet the requirements of Table 1.

The requirements listed in D975 are traditionally based on the assumption that diesel fuel is refined from petroleum crude oil using standard refining processes. Therefore, many of the requirements in D975 are included to describe which part of the barrel of petroleum should be used for diesel fuel. [As will be discussed the next section, the assumption of petroleum as the raw material for diesel fuel means that some final properties of the fuel are assumed or taken for granted. That can lead to problems if a given diesel fuel is manufactured from non-petroleum sources, manufactured by non-traditional methods, or contains significant amounts of non-hydrocarbons.]

Regarding typical petroleum diesel fuel property values and ranges, there are no openly available fuel surveys from which to obtain such information. Conducting such a survey is an expensive and time-consuming task. As such, fuel surveys tend to be limited in scope and/or the results are

confidential. However, since most diesel fuel in the United States is produced to meet ASTM D975, the specific requirements listed in the specification are a reliable source of typical fuel properties. This holds true for fuels from outside the United States that would be subject to specifications other than D975.

### **3.2 FUEL PROPERTIES NOT CURRENTLY LISTED IN ASTM D975**

As mentioned in the previous section, D975-compliant diesel fuels have several properties that are important to the fuel but are not specified in D975. Some of these properties are not in D975 because they are the consequence of making diesel fuel from petroleum and are assumed to be within a range that is acceptable to the intended use. Bulk modulus is one such property. Some of these properties, such as fuel stability, can vary greatly depending on fuel composition, but have not been specified. Still other properties have not been specified for other reasons. A discussion of these properties follows.

#### **3.2.1 Fuel Stability**

As a general definition, fuel stability is the resistance of fuel to change caused by environmental or compositional factors [3]. The property of stability is generally divided into thermal stability, oxidation stability, and storage stability. Thermal stability is the resistance to change when the fuel is heated. Oxidation stability is the resistance to change when the fuel is in an oxidizing environment. Storage stability is the resistance to change while the fuel is in storage. Factors that influence storage stability include presence of water, microbial growth, storage tank linings, ambient temperature, and other contaminants such as rust and dirt. Fuel stability is an important property because changes in the fuel can lead to fuel handling problems (such as plugged filters) and/or engine problems (such as injector deposits).

Depending on their composition, fuels can exhibit poorer thermal stability than oxidation stability or storage stability. The other relationships can also be true. For this reason, it is important to evaluate the full spectrum of fuel stability characteristics of any fuel. At this time, there is no one single test method that evaluates all types of stability, a matrix of testing is required.

### **3.2.2 Density**

Although it is not specified in D975, density is a fundamental physical property of fuel. It can be used, along with other properties, to distinguish light and heavy fuels or fuel fractions. Density is used when converting measured volumes to volumes at standard temperatures. This is important for exchange of ownership transactions. Generally, density is a poor measure of fuel quality although it does have some correlation to energy content.

### **3.2.3 Energy Content**

The energy content, or heat of combustion, is the amount of energy released when the fuel is burned completely. It is directly related to fuel efficiency and vehicle range.

### **3.2.4 Bulk Modulus**

The bulk modulus of a substance measures the substance's resistance to uniform compression. It is defined as the ratio of the infinitesimal pressure increase to the resulting relative decrease of the volume. This is an important property because of the possible effect on metering fuel into the injectors that deliver fuel into the engine's combustion chamber.

### **3.2.5 Boiling Point Distribution**

The boiling point distribution is the measure of the chemical composition of the fuel based on the boiling point of each compound. This property is directly related to flash point, viscosity, low-temperature operability, and combustion. D975 has a single boiling point requirement, temperature at 90% evaporated. Historically, this has been sufficient as a specification because of the “made from” petroleum assumption. Additionally, the specification limits on flash point and viscosity help assure that a petroleum-derived diesel fuel will typically have an acceptable boiling point distribution.

### **3.2.6 Vapor Pressure**

The vapor pressure of diesel fuel is typically negligible at normal storage and use temperatures. A diesel fuel with a vapor pressure above 1 pound per square inch (measured at 38 °C) would



usually be considered contaminated. Besides being an indication of contamination, high vapor pressure could also affect the combustion of the fuel in the engine.

### **3.2.7 Trace Components**

In D975 diesel fuel, the most significant trace component is sulfur owing to the EPA regulations that limit sulfur content in diesel fuels. Some nitrogen-containing compounds may be present but these are not currently regulated. Oxygen-containing compounds are the only other trace component that might be found although these are rare. Biodiesel does contain oxygen but is usually present at levels well above trace. Any other compounds are most likely the result of contamination.

### **3.2.8 Cold Soak Filterability**

The cold soak filterability test [4] was developed in response to problems that occurred with the use of biodiesel at low ambient temperatures. According to the significance and use statement from the method:

“Some substances that are soluble or appear to be soluble in biodiesel (B100) at room temperature will, upon cooling to temperatures above the cloud point or standing at room temperature for extended periods, come out of solution. This phenomenon has been observed in both B100 and BXX blends. These substances can cause filter plugging. This method provides an accelerated means of assessing the presence of these substances in B100 and their propensity to plug filters. B100 biodiesel fuels that give short filtration times are expected to give satisfactory operation of BXX blends at least down to the cloud point of the biodiesel blends.”

The test method is most applicable to fuels made from fats, oils, and greases and should be a requirement for any such fuel. The standard low-temperature operability tests, cloud point, pour point, and cold filter plugging point, should be acceptable for fuels composed only of hydrocarbons. Nonetheless, this test should be part of any evaluation testing of proposed alternative diesel fuels in order to rule out the possible problem.

### **3.2.9 Water Separation Characteristics and Interfacial Tension**

The ability of diesel fuel to shed water during storage and use is a key factor in maintaining fuel quality and also in protecting equipment. There are numerous ASTM test methods to assess this property. The portable separometer method for diesel fuel (DSEP) [5] is commonly used to rate the ability of diesel fuel to release entrained or emulsified water when it passes through fiberglass coalescing material. Measuring the interfacial tension [6] of the fuel has been shown to give a more accurate, and sensitive, indication of the water shedding characteristics of a fuel, especially with regard to water coalescing filters.

### **3.2.10 Compatibility with Petroleum Diesel and Biodiesel**

At this time, petroleum diesel and biodiesel are the only two “fuels” specifically approved under D975. Any proposed new fuel should be tested to demonstrate that it is compatible with petroleum diesel and biodiesel. Blends (50:50) of the new fuel with petroleum diesel and with biodiesel/petroleum diesel blends (B6-B20) should be tested using all the tests in this guidance document. These test fuel blends should meet the requirements of the specification for diesel fuel and for biodiesel/petroleum diesel blends, respectively.

### **3.2.11 Fuel System Deposit-Forming Tendencies**

Currently there exists no reliable test for deposit-forming tendencies other than standard engine testing. Such testing requires large amounts of fuel and long test times so these tests are expensive. A bench-scale test is preferred but none is currently available or widely accepted. There are groups (such as the Coordinating Research Council) working to develop such a test. If such a test is developed and gains industry acceptance it should be used to evaluate proposed alternative diesel fuels.

## **4.0 MATERIALS COMPATIBILITY**

The compatibility of fuel with the materials used in the fuel system is always an important consideration. Because of the over 100 years of experience with petroleum diesel fuel, materials in current fuel systems are assumed to be compatible with the fuel. The introduction of biodiesel

in D975 fuels brought to light some material incompatibilities, mostly elastomers, that have generally been addressed. Any new diesel fuel should be thoroughly tested to determine if there are incompatibilities with currently used fuel system materials. There is no standardized list of materials to evaluate but some materials are sufficiently common to prepare a list for this document as shown in Table 1. Consultation with relevant equipment manufacturers may be required on a case-by-case basis, thereby providing additional materials.

As with the list of materials to be tested, there is no widely recognized testing protocol to use when testing materials. Table 2 contains proposed test parameters.

**Table 1 . Materials for Inclusion in Compatibility Testing**

<b>Elastomers and Plastics</b>	<b>Metals</b>
Buna N	Copper
Fluoroelastomer (FKM)*	Zinc
Nylon	Mild Steel
Teflon	Terne Plate
Fluorosilicone	Brass
	Aluminum

\*ASTM D1418 and ISO 1629. There are different types of FKM as categorized in ASTM D1418 and some will be more resistant than others to various fuel components (like acids in unstable biodiesel).

**Table 2. Proposed Material Compatibility Test Parameters**

<b>Material</b>	<b>Storage Conditions</b>	<b>Evaluation Tests</b>
Elastomers and Plastics	<ul style="list-style-type: none"> <li>• Test fuels include petroleum diesel, biodiesel blend (B20), and 50:50 blends of these two with new fuel, and 100% new fuel</li> <li>• Storage temperature is 40 °C</li> <li>• Storage periods are 1, 2, and 4 weeks</li> </ul>	<ul style="list-style-type: none"> <li>• Tensile strength</li> <li>• Swell</li> <li>• Durometer</li> </ul>
Metals	Same as for elastomers and plastics	<ul style="list-style-type: none"> <li>• Weight loss/gain</li> <li>• Visible signs of corrosion</li> </ul>

## 5.0 ADDITIVES

As the demand for higher quality diesel fuel increases in the market, the use of additives to alleviate problems and/or enhance fuel performance also increases. The advent of non-petroleum diesel fuels has also caused an increase in the use of fuel additives to correct deficiencies in the

fuel. At present, there is no uniform control on the use of additives in commercial diesel fuel. Neither is there a list of approved/disapproved additives. However, the fuel user is always at liberty to request (or even demand) that the fuel seller identify the additives present in the fuel and the concentration of each. The user may also insist that certain additives be included or excluded. Unfortunately, the seller is also at liberty to decline the requests and not sell fuel to that user. For these reasons, fuel additives should always be considered a part of any contract for purchase of diesel fuel.

While there are some regulatory controls on the composition of diesel additives, there are no ASTM, or similar, specification requirements to include or exclude any given additives. In general, diesel additives are considered acceptable as long as they do not degrade the performance of the fuel or cause the fuel to be outside of specification limits. This often results in widespread use of diesel additives for a host of reasons. Because the controls on use, and reporting the use of additives, are weak at best, diesel fuel may be additized several times throughout the distribution system. Sometimes more than once with the same additive. As these various additives mix they can interact. In some cases the interactions are benign; but, in some cases the interactions can have unintended consequences. Additionally, additive chemistries can change and often do, also leading to possible detrimental interactions.

Because of the widespread, uncontrolled use of additives in diesel fuel (and biodiesel) it is not possible to devise a test matrix to cover all possible interactions between a new type of diesel fuel and diesel fuel additives. However, some additives are used more often than others and at least these should be evaluated. Classes of additives that should be evaluated include cetane improver, static dissipater, lubricity improver, and low-temperature operability additives. These additive types encompass numerous additives and there is no practical way to list specific compounds in this document. When it becomes necessary to conduct an additive compatibility evaluation, additive suppliers, fuel suppliers, and other experts should be contacted for names of additives to include in the evaluation. Test fuels used in the evaluation should be petroleum diesel, B5, B20, and proposed new fuel. The diesel, B5, and B20 fuels should each be mixed 50:50 with the new fuel and a given additive at 1X and 2X the maximum recommended

treatment rate. These additive-treated fuels should then be tested per ASTM D6468 at both 90 and 180 minutes. This testing will often reveal fuel/additive incompatibilities. Because D6468 is a short test and requires only a small amount of fuel, it should be possible to design an overall matrix to evaluate the compatibility of most, if not all, of the fuel/additive permutations. It would also be useful to prepare blends as above and test them for the property that is addressed by the additive (such as run a cetane number test when evaluating cetane improver additive or a lubricity test with lubricity additive).

As discussed above, there are few real controls on the use of additives in diesel fuel. As such, it is difficult to control the use of “new” additives for either new diesel fuels or as a new answer to an existing problem. Such new additives should be evaluated, to the extent possible, as discussed earlier in this section.

## **6.0 EXHAUST EMISSIONS**

ASTM does not write specifications to cover exhaust emissions. These are typically covered under federal, state, and local air-quality regulations. Therefore, no specific testing protocols or limits are given in this guidance document. Any proposed new fuel should be tested by itself and in blends with petroleum diesel and biodiesel to confirm that it meets air-quality regulations for its intended use. To the extent that a given air-quality regulation has controls on compositional aspects of diesel fuel (such as total sulfur or total aromatics), these properties should be tested to confirm compliance.

## **7.0 EFFECTS ON LUBRICATING OIL**

Very small amounts of raw or unburned fuel can get into the lubricating oil and oil sump during the normal operation of an engine [7]. Fuel properties such as viscosity, volatility, surface tension/interfacial tension, and bulk modulus can effect fuel combustion thereby affecting droplet size, cylinder wall impingement, and, ultimately, fuel dilution from unburned fuel. A first

indication of the possibility of lubricating oil effects with a new fuel can come from comparison of the properties listed above with the same property values from petroleum fuel and biodiesel blends. New fuels with significantly different viscosity, volatility, etc., may be more likely to cause fuel dilution of the engine lubricating oil.

For more detailed/precise assessments of fuel/oil interactions, actual engine testing would be required. The fuel should be tested in the engine used in the expected application. In the case of several different engines, an attempt should be made to choose engines with known sensitivity to fuel dilution.

## **8.0 HANDLING AND DISTRIBUTION**

In order to be considered a drop-in replacement fuel, any proposed new fuel must be compatible with existing fuel handling and distribution infrastructure. These fuels must also be no more dangerous to the persons using the fuel when compared to conventional diesel fuel. This includes both direct contact with the fuel and inhalation of fuel engine exhaust.

The first source of information regarding the hazards associated with a new fuel is the safety data sheet, SDS, for the new fuel. The SDS identifies any hazards related to the use and handling of the fuel. A typical SDS has the following information:

- Hazards Identification
- Composition
- First Aid Measures
- Fire Fighting Measures
- Accidental Release Measures
- Handling and Storage
- Exposure Controls / Personal Protection
- Physical and Chemical Properties
- Chemical Stability and Reactivity

- Toxicological Information
- Ecological Information
- Disposal Considerations/Information
- Transportation Information
- Regulatory Information

Compositional data for a new fuel will also provide valuable information regarding use and handling of the fuel. Comparison of new fuel composition with that of existing D975 fuels can be an indication of possible concerns.

Laboratory testing to measure relevant properties such as corrosivity, flash point, water separability, etc., is also important to assess the use and handling aspects of a new fuel. The EPA alternative fuels registration process requires that the producer of a new fuel provide information regarding the product emissions.

## **9.0 OVERVIEW OF U.S. EPA ALTERNATIVE FUELS REGISTRATION PROCESS**

Under section 211 of the Clean Air Act, refiners and importers must register their products with EPA before those products are offered for sale. EPA uses registration information to identify product emissions that may pose an unreasonable risk to public health. In certain cases, health effects testing is required before a new product can be registered, or for an existing product to maintain its registration.

Only gasoline and diesel fuel and fuel additives produced and commercially distributed for use in highway motor vehicles must be registered. Fuel intended for use exclusively in off-road vehicles, engines, or equipment is not required to be registered.

Under the registration program, manufacturers are required to analyze the combustion and evaporative emissions generated by their fuel and fuel additive products, survey existing scientific information for each product and, where adequate information is not available, conduct tests to screen for potential adverse health effects of these emissions. The registration regulations may be found at Title 40 CFR Part 79.

## **10.0 THE USE OF FUEL COMPOSITION TO ESTIMATE/PREDICT FUEL PERFORMANCE AND ACCEPTABILITY**

Using fuel composition to predict (estimate) fuel performance and acceptability has been done for years. Some examples include:

- Using fuel composition to estimate the cetane number of diesel fuel (ASTM D976 and ASTM D4737).
- Using cetane index as a control on fuel aromaticity. [8]
- Controlling total sulfur in order to prevent damage to engine emissions after-treatment devices.
- Using infrared spectroscopy to estimate fuel composition and properties [9] [10] [11].
- Several ASTM methods for estimation of net heat of combustion of aviation fuels.

The main problem with estimation and prediction methods is that they often rely on previously determined correlations of fuel properties and fuel composition information. The correlations may work very well for the specific properties and fuels in the original correlation study; but, that does not guarantee similar success with other fuels. The cetane index method, D4737, works well for estimating cetane number of petroleum diesel but is not applicable to fuel containing biodiesel.

Morris and co-workers [12] have demonstrated the use of 2-dimensional gas chromatography/mass spectrometry to provide valuable fuel compositional data. These data can then be used in further studies and correlations with the fuel.



Any use of fuel compositional data to estimate or predict the performance of a new fuel must be done with considerable use of technical judgment. Accurate modeling/correlations require robust studies which are typically not available with new fuels. However, to the extent that reliable information is available and correlations to performance properties are reliable, using them to estimate performance properties can be a useful technique.

## **11.0 PERSPECTIVES FROM STAKEHOLDERS**

The Truck and Engine Manufacturers Association is a trade organization for their industry and publishes position statements for the industry. One such position statement is titled “Facts You Should Know About Renewable Fuels”, dated October 2009. It contains several statements regarding new and renewable diesel fuels as:

- Engine manufacturers are neutral on the feedstock used to produce biomass-based diesel fuels. The critical performance factors of any diesel fuel — petroleum-based or biomass-based — are derived from the end product and not on the source or characteristics of the feedstock. In the United States and around the world, there is an emerging interest in non-ester renewable diesel fuel. Nonetheless, the vast majority of biomass-based diesel fuel currently being produced or planned is biodiesel. Therefore, operational experience with non-ester renewable diesel fuels is very limited.
- Poor-quality fuel that does not meet internationally recognized standards is not acceptable at any blend level in that it can prevent the engine from starting or result in damage to engine and/or emission control components, loss of power or fuel efficiency, and excess emissions. Similarly, raw, unprocessed vegetable oils and animal fats, regardless of blend level, can have significant adverse effects and should not be used as fuel in diesel engines. See EMA’s Statement regarding the “Use of Raw Vegetable Oils or Animal Fats in Diesel Engines.”

- Engine manufacturers have limited experience with renewable diesel fuel or renewable diesel fuel blends. These fuels are expected to provide acceptable performance due to their petroleum-like properties, but lack of field experience to confirm expectations requires that engine manufacturers remain cautious with respect to making any recommendations for these blends.
- Non-esterified renewable diesel fuels with essentially the same properties as conventional paraffinic diesel fuel show great promise. These second generation renewable diesel fuels eliminate some of the properties that are of concern with biodiesel. While their cold flow properties and energy content must be considered carefully, they deserve additional development and study.
- In every case, owners and operators of compression ignition engines should refer to their engine manufacturer's fuel recommendations prior to making any decisions regarding use of an alternative fuel. Use of unprocessed oils, greases, or fats may result in reduced engine life, increased maintenance costs, or catastrophic engine failure. The potential adverse effects of these oils, greases, and fats increase as fuel injection system pressure increases and sophisticated injection strategies become more prevalent. Moreover, the problems associated with their use, regardless of blend level, may not become evident until a significant amount of damage has occurred over an extended period.

## **12.0 SUGGESTED TESTING PROTOCOL FOR ALTERNATIVE DIESEL FUELS**

The following information/test results should be provided along with any request to designate a new fuel as a drop-in alternative diesel fuel. Each request should be taken on a case-by-case basis since comparison of this information with current D975 fuels can be different depending on the new fuel.

- Must meet all the requirements of ASTM D975.
- Must be registered under the EPA Alternative Diesel Fuel Registration Process.

- Must have been tested for the following properties and report the results:
  - Oxidation stability
  - Thermal stability
  - Storage stability
  - Density
  - Energy Content
  - Bulk Modulus
  - Complete boiling point distribution
  - Vapor pressure at 38 °C
  - Heteroatomics content
  - Metals content
  - Hydrocarbon types by ASTM D2425 or 2-dimensional GC/MS
  - Cold soak filterability
  - Water separation characteristics and interfacial tension
  - Compatibility with petroleum diesel and biodiesel
  - Fuel system deposit formation if and when a test method becomes available
  - Fuel system materials compatibility
  - Additive compatibility
- Report any additives routinely used in the new fuel
- Provide a complete safety data sheet for the new fuel

### 13.0 REFERENCES

1. "Standard Specification for Diesel Fuel Oils," ASTM D975, ASTM International, 100 Barr Harbor Drive, PO Box C700, West Conshohocken, PA 19428-2959.
2. "Standard Specification for Diesel Fuel Oils," ASTM D975, ASTM International, 100 Barr Harbor Drive, PO Box C700, West Conshohocken, PA 19428-2959.
3. Distillate Fuel Stability and Cleanliness, ASTM Special Technical Publication 751, Stavinocha and Henry, eds., ASTM, 1981.
4. Standard Test Method for Determination of Fuel Filter Blocking Potential of Biodiesel (B100) Blend Stock by Cold Soak Filtration Test (CSFT), ASTM D7501.
5. Standard Test Method for Determining Water Separation Characteristics of Diesel Fuels by Portable Separometer, ASTM D7261
6. Standard Test Methods for Surface and Interfacial Tension of Solutions of Surface-Active Agents, ASTM D1331.
7. Fang, H., Whitacre, S., Yamaguchi, E., and Boons, M., "Biodiesel Impact on Wear Protection of Engine Oils," SAE Technical Paper 2007-01-4141, 2007, doi:10.4271/2007-01-4141.
8. 40 CFR Part 80 (See Table 1, ASTM D975)
9. The Use of AOTF-NIR Spectrometers to Analyze Fuels – Phase I: Instrument Selection and Preliminary Calibrations, TFLRF Report No. 313
10. Estimation of Middle Distillate Fuel Properties by FT-IR and Chemometrics – Part I: Calibrations and Validations, TFLRF Report No. 321.
11. Estimating Chemical and Bulk Properties of Middle Distillate Fuels from Near-Infrared Spectra, TFLRF Report No. 348
12. Morris, R.E., et.al., "Development of a Compositional Definition of Jet and Diesel Fuels Using a Novel Automated Compound Classification Scheme," Proceedings of the 12<sup>th</sup> International Conference on Stability, Handling, and Use of Liquid Fuels," 16-20 October, 2011.